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**(54) FILTER MATERIAL HAVING PHOTOCATALYSIS, ITS PRODUCTION AND PHOTOCATALYTIC FILTER**

(57)Abstract:

PROBLEM TO BE SOLVED: To obtain a high performance filter having photocatalysis at a low cost by partially coating a filter substrate with protrusions formed on the surface and particles for forming the protrusions with a photocatalyst or a photocatalyst-contg. material and bonding these together by the photocatalyst or photocatalyst-contg. material.

SOLUTION: The material of the filter substrate is, e.g. glass, ceramics, glass ceramics, a metal, a metallic mesh, plastics or crystals but transparent glass, ceramics, plastics or crystals are preferably used. Titanium dioxide is suitable for use as the photocatalyst from the viewpoint of photocatalytic activity and profitableness. Particles of ceramics, glass, glass ceramics, a metal or plastics may be used as the particles for forming the protrusions.

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**CLAIMS**

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[Claim(s)]

[Claim 1]Are the filter material in which a projection was formed in the surface, and while at least a part is covered with a photocatalyst or a photocatalyst inclusion, a filter base material and particles for projection formation, A filter material, wherein the filter base material concerned and the particle for projection formation concerned are combined by a photocatalyst or photocatalyst inclusion.

[Claim 2]The filter material according to claim 1 whose filter base material is glass fiber.

[Claim 3]The filter material according to claim 1 or 2 a photocatalyst or whose photocatalyst inclusion is a titanium dioxide or a titanium dioxide inclusion.

[Claim 4]When covering a photocatalyst or a photocatalyst precursor to a filter base material and particles for projection formation and using a photocatalyst precursor, A manufacturing method of a filter material performing simultaneously a process of including operation of furthermore changing it into a photocatalyst, and a process which combines the particles for projection formation concerned with the filter base material concerned.

[Claim 5]A photocatalyst filter which uses the filter material according to claim 1, 2, or 3.

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## DETAILED DESCRIPTION

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[Detailed Description of the Invention]

[0001]

[Field of the Invention]This invention relates to the filter material which has photocatalyst ability, its manufacturing method, and a photocatalyst filter. It is related with the photocatalyst filter using the raw material of the photocatalyst filter which is used suitably for water treatment, an environmental clean-up, etc. as for this invention, the method of manufacturing this thing efficiently, and the still more detailed above-mentioned filter material.

[0002]

[Description of the Prior Art]The filter of three sorts of types generally shown below when purifying a gas and a fluid through a filter conventionally, Namely, the filter which filled up with (1) staple-fiber fiber the space which forms a filter. The filter using what carried out the spinning and weaving of (it is hereafter called a staple fiber filter) and the (2) continuous-glass-fiber fiber, and was made blanket-like as a filter material. The filter which fabricates (it being hereafter called a continuous glass fiber filter) and (3) granular material, and consists of a sintered porous body, or the filter which filled up with granular material the space which forms a filter is used.

[0003]However, although adjustment of the hole (mesh) of a filter is performed by mainly changing the pack density of a fiber in the above-mentioned staple fiber filter, When it was easy to produce a density difference by a place, and exact control of a mesh is difficult and also it lets a fluid pass, the shape of a mesh changes easily, therefore there is a problem that a highly efficient filter is hard to be obtained.

[0004]In a continuous glass fiber filter, weave a continuous glass fiber fiber and the complicated process of using blanket-like is needed, It does not escape becoming a high cost, and also the diameter of a fiber in which spinning and weaving are possible has a limit, and a realizable mesh is restricted, therefore there is a problem that a highly efficient filter is hard to be obtained.

[0005]On the other hand, although granular material may be fabricated by methods, such as sintering, and what was used as the porous body may be used as a filter in the filter of the type of the above (3), In this case, the space where granular material is required for a large

quantity, and it is big is required, and also cost starts shaping and there is a problem that it does not escape that a manufacturing cost costs dearly.

[0006]Thus, the actual condition was that each filter of the conventional type is difficult to have a certain fault and to provide a highly efficient filter cheaply.

[0007]Then, a group including this invention person repeats research wholeheartedly that a highly efficient filter should be provided cheaply, It found out that a highly efficient filter could be provided cheaply by developing previously a filter material new type [ in\_which the projection was formed on the surface of the filter material ], and using this filter material (Japanese Patent Application No. No. 248843 [ eight to ]).

[0008]By the way, it is known that a certain kind of metallic compounds have a photocatalyst effect. Since this photocatalyst effect is absorbing light energy, ionization oxygen molecules, such as a superoxide anion ( $-O_2^-$ ) and a hydroxy radical ( $-OH$ ), are generated and oxidative degradation of the organic matter is carried out as a result, In recent years, the trial which it is going to apply to a filter at various water treatment and air processing, an environmental clean-up, etc. using this photocatalyst effect is performed positively.

[0009]As metallic compounds (a photocatalyst may be called hereafter) which have a photocatalyst effect, For example, a titanium dioxide, barium titanate ( $BaTi_4O_9$ ), Although strontium titanate ( $SrTiO_3$ ), titanium sodium ( $Na_2Ti_6O_{13}$ ), a zirconium dioxide, a cadmium sulfide,  $\alpha-Fe_2O_3$ , etc. are known, a titanium dioxide is typical in these.

[0010]Since a group including this invention person provided a highly efficient filter cheaply as mentioned above, proposed the filter material in which the projection was formed on the surface of the filter base material, but. Under the present circumstances, in order to provide cheaply the highly efficient filter which has photocatalyst ability, the filter material which made the projection form on the surface of a filter base material, and made this substrate support a photocatalyst further was proposed.

[0011]However, it is necessary to provide a photocatalyst layer in the base material surface in which the projection was provided in this case anew, and operation is complicated and it may not necessarily be satisfied [ operation ] fully in respect of a process.

[0012]

[Problem(s) to be Solved by the Invention]This invention is a basis of such a situation and an object of this invention is to provide the filter material which can provide cheaply the highly efficient filter which has the photocatalyst ability used suitably for water treatment, an environmental clean-up, etc., its manufacturing method, and the photocatalyst filter using the above-mentioned filter material.

[0013]

[Means for Solving the Problem]In order that this invention person may attain said purpose, as a result of repeating research wholeheartedly, by photocatalyst or a photocatalyst inclusion. By combining both at the same time it covers a filter base material and particles for projection formation, That a filter material in which a projection layer with photocatalyst ability was formed

is obtained easily, and by using this filter material, it finds out that a highly efficient filter which has photocatalyst ability is obtained cheaply, and came to complete this invention based on this knowledge.

[0014]Namely, this invention is the filter material in which a projection was formed in the surface, and while at least a part is covered with a photocatalyst or a photocatalyst inclusion, a filter base material and particles for projection formation, A filter material in which the filter base material concerned and the particles for projection formation concerned are characterized by being combined by a photocatalyst or photocatalyst inclusion, and a photocatalyst filter which uses this filter material are provided.

[0015]If this invention is followed, said filter material, When covering a photocatalyst or a photocatalyst precursor to a filter base material and particles for projection formation and using a photocatalyst precursor, it can manufacture by performing simultaneously a process of including operation of changing it into a photocatalyst further, and a process which combines the particles for projection formation concerned with the filter base material concerned.

[0016]

[Embodiment of the Invention]There is no restriction in particular about the construction material of the filter base material used in the filter material of this invention, and arbitrary things can be chosen and used out of what is commonly used as a filter base material conventionally. As construction material of this substrate, although glass, ceramics, crystallized glass, metal, a metallic mesh, plastics, a crystal, etc. are mentioned, for example, in order to make a photocatalyst effect reveal effectively, the thing of a light transmittance state, for example, glass, ceramics, a plastic, a crystal, etc. are preferred. This filter base material may consist of an independent material, \*\*\*\* is also good, it may mix or compound and two or more sorts of materials may be used (for example, junction etc.).

[0017]It is preferred that it is what does not react to the photocatalyst itself as a filter base material of a light transmittance state. It is more preferred that it is a substrate which does not lower photocatalyst activity.

[0018]While there is no diffusion of the impurity to the inside of a photocatalyst and not degrading photocatalyst activity as a substrate which does not lower photocatalyst activity, it is easy to form a photocatalyst thin film, and excels in chemical durability, transparency, etc., and what has possible forming in continuous glass fiber is mentioned.

[0019]As such a substrate, the silicate glass of low alkali, the aluminosilicate glass, borosilicate glass, or alkali free glass whose content of 30 to 70% and an alkali component it is a weight % display and is 0 to 10% about  $\text{SiO}_2$  is mentioned, for example.

[0020]Although there is no restriction in particular as shape of a filter base material, for example, publicly known shape, such as fibrous, tabular, the shape of a rod, the shape of a bead, blanket-like, and the shape of granular material (a porous body is included), can be mentioned, [ which can obtain a filter cheaply only by only bundling a filter material or laminating / a point to / the fibrous or tabular ] is advantageous.

[0021]As this filter base material, the glass fiber for photocatalyst support is especially preferred.

[0022]In order to make a photocatalyst effect reveal effectively, a light guide nature substrate can be used. In this case, as a material, the aforementioned light transmittance state substrate, for example, glass, ceramics, plastics, a crystal, etc. are used. Such materials may be used independently, it may mix or compound and two or more sorts may be used (for example, junction etc.). As shape of this light guide nature substrate, the shape of a fiber, honeycomb shape, mesh shape, blanket-like, stratified, the shape of cotton, etc. are mentioned, for example. The shape of this light guide nature substrate may be a single configuration, and two or more sorts of shape may be composite-ized (for example, junction etc.).

[0023]Since the light which entered that a light guide nature substrate was a fiber-like from one end of the fiber leaks from the side little by little and is transmitted in the inside of a fiber with appearance, the photocatalyst formed in the surface can be irradiated efficiently.

[0024]As a photocatalyst used in the filter material of this invention, What is conventionally publicly known, for example, a titanium dioxide, strontium titanate ( $\text{SrTiO}_3$ ), Barium titanate ( $\text{BaTi}_4\text{O}_9$ ), titanium sodium ( $\text{Na}_2\text{Ti}_6\text{O}_{13}$ ), A zirconium dioxide, a cadmium sulfide,  $\alpha\text{-Fe}_2\text{O}_3$ ,  $\text{K}_4\text{Nb}_6\text{O}_{17}$ ,  $\text{Rb}_4\text{Nb}_6\text{O}_{17}$ ,  $\text{K}_2\text{Rb}_2\text{Nb}_6\text{O}_{17}$ ,  $\text{Pb}_{1-x}\text{K}_{2x}\text{Nb}_2\text{O}_6$ , etc. are mentioned. Although these may be used independently and it may use combining two or more sorts, the titanium dioxide in these is preferred from the field of photocatalyst activity and economical efficiency. Although this titanium dioxide has a rutile type, an anatase type, etc., the anatase type is more preferred than a rutile type from a photocatalyst activity point.

[0025]On the other hand, the particles for projection formation used in the filter material of this invention do not have restriction in particular as the shape, for example, a globular shape, unfixed shape, rod form, the shape of a scale, fibrous, a porous body, etc. are mentioned. As construction material, ceramics, glass, crystallized glass, metal, plastics, crystals (alumina, zirconia, a titania, mullite, Caux Delight, magnesia, barium titanate, etc.), particles (particles of glassiness and a crystalline substance, etc.), etc. are mentioned, for example.

[0026]Although one sort of these particles for projection formation may be used and it may use combining two or more sorts, especially a glass bead is preferred. Although it changes with shape of this particle and cannot generally set, when [ , for example like a glass bead ] spherical, the size of the particles for projection formation has a preferred thing of the range of 0.1-1,000-micrometer mean particle diameter, and its thing of the range which is 2-200 micrometers is especially preferred.

[0027]There is no restriction in particular about the distribution density of a projection, and it is suitably selected in consideration of the degree of hole (mesh), the pressure of pressure loss and a fluid and quantity and the intensity of a substrate, a path and thickness, collection efficiency, etc.

[0028]As for the filter material of this invention, they are combined while a filter base material and the particles for projection formation are covered with an aforementioned photocatalyst or photocatalyst inclusion.

[0029>About the manufacturing method of such a filter material. If the method of this invention

is followed, when covering a photocatalyst or a photocatalyst precursor to the aforementioned filter base material and the particles for projection formation and using a photocatalyst precursor, A desired filter material can be manufactured by performing simultaneously the process of including operation of furthermore changing it into a photocatalyst, and the process which combines the particles for projection formation with the above-mentioned filter base material.

[0030]In this case, the coating liquid which makes a photocatalyst and the particles for projection formation come to be suspended in two sorts of methods, i.e., (1) binder solution, specifically, How to make the projection layer which applies to the surface of a filter base material, dries it, and has photocatalyst ability form, (2) Into the binder (photocatalyst precursor) solution which demonstrates photocatalyst ability by heat treatment, heat treatment is performed after applying to the surface of a filter base material the coating liquid which makes the particles for projection formation come to be suspended, and the method of making a projection layer with photocatalyst ability form is used.

[0031]First, the 1st method [method of (1)] of the above is explained.

[0032]As a binder solution in this 1st method, the solution which dissolved various binders in the suitable solvent is used. As a binder, for example Under the present circumstances, the alkoxide compound of silicon, silicone resin, In addition, a silicon content organic compound, the alkoxide compound of a zirconium and other zirconium content organic compounds, the alkoxide compound of aluminum and other aluminum content organic compounds, an alkoxide compound, other organic titanium compounds of titanium, etc. are mentioned.

[0033]Here as an example of the alkoxide compound of silicon, The oligomer, such as tetraethyl orthosilicate, tetramethyl orthosilicate, and methyl triethoxysilane, as an example of silicone resin, In addition to this, methyl silicone resin, phenyl silicone resin, methylphenyl silicone resin, etc. are mentioned for the oligomer, such as methylsilyl tri-isocyanate, as an example of a silicon content organic compound. As an example of the alkoxide compound of a zirconium, or other zirconium content organic compounds, The oligomer, such as zirconium tetra-n-propoxide and zirconium tetra-n-butoxide, The oligomer, such as zirconium acetate and zirconium acetylacetonate, is mentioned, As an example of the alkoxide compound of aluminum, or other aluminum content organic compounds, the oligomer, such as the oligomer, such as aluminum tri-sec-butoxide, and aluminium diisopropoxide ethylacetoacetate chelate, is mentioned.

[0034]As an example of the alkoxide compound of titanium, or other organic titanium compounds, Titanium tetra-n-butoxide, titanium tetra isopropoxide, Titanium tetraethoxide, tetra(2-ethylhexyloxy) titanium, Tetrastearoxy titanium, diisopropoxybis(acetylacetonate) titanium, The oligomer, such as the oligomer, dihydroxybis(RAKUTATO)titanium, titanium stearate, etc., such as di-n-butoxy(triethanol AMINATO) titanium and titanium isopropoxy octylene glycolate, is mentioned.

[0035]These binders may be used independently and may be used combining two or more sorts. As a solvent used for preparation of a binder solution, While dissolving said binder, it has volatility, and desiccation of a coat should be just easy, and is not restricted in particular, but

various organic solvents, for example, an alcohol system, an ester system, a ketone system, an ether system, etc. can be used. These solvents may be used independently, and two or more sorts may be mixed and they may be used. What is necessary is just to select suitably about the binder concentration in a binder solution in consideration of spreading nature, handling nature, etc. of coating liquid which are obtained by there being no restriction in particular.

[0036]Although the thing of the above mentioned kind is mentioned as a photocatalyst added to this binder solution and that shape is not asked, especially the thing of particle shape is preferred.

[0037]These photocatalyst particulates have a preferred thing in the range of 5-1,000-nm mean particle diameter.

[0038]On the other hand, as particles for projection formation made suspended in a binder solution, the thing of the kind mentioned above and shape can be used.

[0039]After making said photocatalyst of the specified quantity, and the particles for projection formation suspended homogeneously and preparing coating liquid in a binder solution in the 1st method, This coating liquid is applied on the surface of a filter base material by the publicly known method, for example, a dipping method, the flow-coating method, the spraying method, etc., and a drying process is carried out further. This drying process is performed by usually heating at the temperature of about 50-200 \*\*.

[0040]Thus, the projection layer in which thickness has 0.01-40 micrometers of photocatalyst ability which is 0.1-20 micrometers preferably especially preferably is formed.

[0041]The reason made preferred [ 0.01-40 micrometers ] as projection layer thickness has an insufficient quantity of a photocatalyst, or the absorption of light is [ case below 0.01 micrometer ] insufficient for it, It is because there is a possibility that a crack may go into a film easily and exfoliation may arise from a crack when there is a possibility that an efficient photocatalyst effect cannot be caused and it exceeds 40 micrometers on the other hand.

[0042]Thus, the abundance of the photocatalyst particulates and the particles for projection formation in the projection layer which has photocatalyst ability which were formed, and a binder, 1-85:5-85:10-90 are preferred, 5-85:5-85:10-90 are more preferred, and 10-70:10-70:20-especially 80 are preferred at a weight ratio (total 100).

[0043]Next, the 2nd aforementioned method [method of (2)] is explained.

[0044]In this 2nd method, the solution which contains the binder (photocatalyst precursor) which reveals photocatalyst ability by heat treatment as a binder solution is used. Here, as a binder which reveals photocatalyst ability by heat treatment, organic titanium compounds, such as an alkoxide compound of titanium, etc. are used, for example.

[0045]As an example of the alkoxide compound of said titanium, Titanium tetra-n-butoxide, titanium tetra isopropoxide, Titanium tetraethoxide, tetra(2-ethylhexyloxy) titanium, Tetrastearoyloxy titanium, diisopropoxybis(acetylacetonate)titanium, The oligomer, such as di-n-butoxy(triethanol AMINATO) titanium and titanium isopropoxy octylene glycolate, is mentioned, As an example of other organic titanium compounds, the oligomer, such as dihydroxybis (RAKUTATO)titanium and titanium stearate, is mentioned. By heat treatment of about 400-700



\*\*, the crystal of an anatase type titanium dioxide deposits and these titanium compounds reveal a photocatalyst effect. If heat treatment temperature is too high not much, a rutile titanium dioxide comes to be formed and a mentioned range is suitable undesirably.

[0046]These titanium compounds may carry out quantity concomitant use of the binders other than the titanium compound which could be used independently, and could be used combining two or more sorts, or was illustrated in other binders, for example, said 1st method, if needed suitably.

[0047]As a solvent used for preparation of a binder solution, what is necessary is to have volatility, while dissolving said binder, and not to just be restricted in particular but, and various organic solvents, for example, an alcohol system, an ester system, a ketone system, an ether system, etc. can be used. These solvents may be used independently, and two or more sorts may be mixed and they may be used. What is necessary is just to select suitably about the binder concentration in a binder solution in consideration of spreading nature, handling nature, etc. of coating liquid which are obtained by there being no restriction in particular.

[0048]As particles for projection formation distributed in this binder solution, the thing of the kind mentioned above and shape can be used like said 1st method.

[0049]In this 2nd method, in the binder solution which reveals photocatalyst ability by heat treatment, After distributing the particles for projection formation of the specified quantity homogeneously and preparing coating liquid, this coating liquid is applied on the surface of a filter base material by the publicly known method, for example, a dipping method, the flow-coating method, the spraying method, etc. Subsequently, after carrying out a drying process at the temperature of about 50-200 \*\* if needed, the projection layer in which thickness has 0.01-40 micrometers of photocatalyst ability which is 0.1-20 micrometers preferably especially preferably is formed by performing heat treatment of about 400-700 \*\*.

[0050]Thus, it is a weight ratio (total 100), as for the abundance of the particles for projection formation and binder (photocatalyst) in a projection layer with photocatalyst ability which were formed, 1-90:10-99 are preferred, and 5-80:20-especially 95 are preferred.

[0051]In this 2nd method, in preparation of coating liquid, in order to promote hydrolysis reactions, such as the binder which reveals photocatalyst ability by heat treatment, for example, the alkoxide compound of titanium, etc., it is usually adjusted by acid, such as chloride, so that pH may be on an acidity side.

[0052]They are the purposes, such as raising photocatalyst ability or improving the adhesion over the substrate of a projection layer in said 1st and 2nd methods, For example, Ag, Cu, Au, Pt, Ru, Pd, Rh, Sn, Si, In, The powder of metal, such as Pb, As, and Sb, and compounds, such as the oxide, The coated layer which may add in coating liquid by request, and contains compounds, such as the above-mentioned metal and its oxide, beforehand on the surface of a filter base material is formed, on it, coating liquid may be applied, desiccation or heat treatment may be performed, and a projection layer may be provided.

[0053]Thus, the filter material in which the projection layer which has photocatalyst ability in a base material surface was provided is obtained efficiently.

[0054]The parameter with this preferred projection layer is the same as that of what was

obtained by the 1st method of the above.

[0055]This invention also provides the photocatalyst filter which uses again the filter material obtained by the aforementioned method.

[0056]In order to constitute a filter using a filter material, the publicly known filter mode according to the shape of the filter material, etc. can be used.

[0057]For example, when it constitutes a filter as a filter material using the fiber which provided the projection layer in the surface, a filter with a necessary crevice (mesh) can be formed only by bundling two or more fibers to a uniform direction mostly, and only fixing. Under the present circumstances, the granularity of a mesh is arbitrarily changeable by the size of a projection, an interval, distribution density, etc.

[0058]When it constitutes a filter as a filter material using the substrate which provided the projection layer in the surface, a filter with a necessary crevice (mesh) can be formed only by laminating two or more substrates and fixing. Under the present circumstances, the granularity of a mesh is arbitrarily changeable by the interval of the size of a projection, an interval, distribution density, and a substrate, etc.

[0059]In this invention, it can be considered as the transparent material which draws the light of wavelength required in order to make a photocatalyst into an active state for a filter material, the light drawn by the transparent material can come out from the transparent material surface, and it can also be considered as the filter it was made to reach a photocatalyst. Under the present circumstances, as shape of a transparent material, as described above, for example, the shape of a fiber, honeycomb shape, mesh shape, blanket-like, stratified, the shape of cotton, etc. may be any. This shape may be a single configuration and may composite-ize two or more sorts of shape.

[0060]Light can be introduced into the photocatalyst formed on the surface of the filter material only by bundling this as a filter material as a transparent material is a fiber-like, making it laminate, and entering light from one end.

[0061]Selection of a photocatalyst and transparent material material is good to take both refractive index into consideration. This is for making light leak to the photocatalyst layer side which is covering material unlike the optical fiber which confines light in a core, and it is advantageous to choose a photocatalyst with a big refractive index compared with transparent material material.

[0062]The filter material of this invention has the following effects on the fiber which formed the projection beforehand as compared with the filter material which covered the photocatalyst.

[0063](i) Since the projection is not formed in the fiber itself, the filter material of this invention does not have a thing of a projection for which lights are scattered about by the way like [ at the time of making the fiber surface unevenness and forming a projection ]. As a result, it can use efficiently to the end of a filter material.

[0064](ii) Since considering the case where the projection of the same size is attached the way which the projection has not attached to a transparent material beforehand can make a photocatalyst exist also between a projection and a transparent material, more photocatalysts can be attached. Photocatalyst capability can be improved if the quantity of a photocatalyst

increases.

[0065](iii) Compared with the case where a projection is formed beforehand, manufacture is easy for a fiber.

[0066]In the filter device equipped with the photocatalyst filter of this invention, in order to make a photocatalysis perform in a projection layer with the photocatalyst ability provided in the base material surface, a light source is installed so that the light of wavelength required in order to make a photocatalyst this projection layer at an active state may be irradiated. As light source wavelength, when a photocatalyst is a titanium dioxide, the 200-500-nm ultraviolet rays which can excite this are preferred, and the mercury lamp and ultraviolet ray lamp which output this with continuation light can be used.

[0067]In the photocatalyst filter of this invention, a photocatalysis is caused on the surface, strong oxidizing power and reducing power are produced, and the substance caught by the filter is disassembled and removed. As a substance in which catching in the gas which can carry out decomposition removal is possible, For example, the smoke of fumes, dust, air dust, and tobacco, dust, a virus, bacteria, malodorous substances (acetaldehyde, methyl mercaptan, etc.), etc. are mentioned, and sludge, an organic matter, trihalomethane, etc. are mentioned as a substance contained in a solution, for example.

[0068]The filter device equipped with the photocatalyst filter of this invention, For example, the diesel particulate filter (DPF) for removing the solid granular material (particulate) which consists of a black smoke contained in the exhaust gas of a diesel power plant, and unburned hydrocarbon and a lubricating oil, It can be conveniently used as a gas treatment filter (for example, the air filter for clean rooms, an air cleaner), a liquid treatment filter (for example, water and the filter for sea water purification), etc.

[0069]

[Example]Next, although an example explains this invention still in detail, this invention is not limited at all by these examples.

[0070]Example 1 (the 1st method)

After dissolving 1.50 kg of methyl silicone resin in 8.50 kg of isobutyl acetate, and a mixed solution with 17.00 kg of isopropanol, 1.5 kg of anatase type titania ultrafine particles with a mean particle diameter of 20 nm were added, and it was made to distribute using BISUMIRU. 1.5 kg of glass BISU with a mean particle diameter of 15 micrometers was made suspended to these dispersion liquid, and 30 kg of coating liquid was prepared.

[0071]Next, the projection layer which applies the above-mentioned coating liquid to the surface, dries on it, and has photocatalyst ability in it was made to form, lengthening glass fiber using the device shown in drawing 1, as shown below.

[0072]The glass fiber 3 by which drawing 1 is a schematic diagram of the device for making the projection layer which has photocatalyst ability on the surface of a fiber form, and spinning was carried out with the spinning device 1, After the coating liquid supplied to the surface from the coating liquid feeding device 2 is applied, the projection layer which a drying process is carried out at 150 \*\* with the heating furnace 4, and has photocatalyst ability is formed, and, subsequently it is rolled round by the winding roll 5. The figure with which (a) looked at the

coating liquid application part from the top, and (b) are the figures seen from width.

[0073]thus, in the obtained filter material, the average thickness of 125 micrometers and a projection layer of the path (before the projection stratification and the following -- the same) of glass fiber was 16 micrometers. When the fiber surface was investigated by microscope observation, it turned out that many projections are formed. Although this filter material was strongly wiped away with the "KIMUWAIPU wiper S200" (made in 10 \*\* Kimberley, trade name) which carried out humidity by isopropanol, the projection did not exfoliate.

[0074]Drawing 2 is a microphotograph of the glass fiber (2) in which the projection layer was provided on the surface of the above. The microphotograph of the glass fiber (2) in which the projection layer is not formed for reference is shown in drawing 3.

[0075]The solidified body of the above-mentioned coating liquid was produced, and the X diffraction was performed about what heat-treated at 150 \*\*. The chart is shown in drawing 4.

[0076]Example 2 (the 1st method)

1.0 kg of glass beads with a mean particle diameter of 10 micrometers were made suspended to 29.0 kg of photocatalyst-titanium-oxide coating liquid (Ishihara techno company make and trade name:ST-K03, anatase type titania, 5 % of the weight of particle dispersion liquid), and coating liquid was prepared to it.

[0077]After applying to a glass fiber bundle (30 micrometers x about 10,000 diameters) with a dip coating method, having put this coating liquid into a cylindrical container the diameter of 100 mm, and 700 mm in length, and carrying out ultrasonic impression, it heat-treated at 150 \*\* and the projection layer with photocatalyst ability was formed.

[0078]Thus, in the obtained filter material, the average thickness of the projection layer was 11 micrometers, and when the surface was investigated by microscope observation, it turned out that many projections are formed. Although strongly wiped away by KIMUWAIPU to which humidity of this filter material was carried out by isopropanol, the projection did not exfoliate.

[0079]Example 3 (the 2nd method)

Agitating the mixed solution of 3.76 kg of acetylacetones (stabilizing agent), and 8.84 kg of isopropanol, 6.39 kg of titanium tetra-n-butoxide was added and it mixed overnight. After adding gradually the hydrochloric acid water 677g of 0.15-mol [l. ] concentration, and a mixed solution with 8.84 kg of isopropanol to this solution and agitating them in it for 3 hours, 1.5 kg of glass beads with a mean particle diameter of 10 micrometers were made suspended to this, and 30 kg of coating liquid was prepared.

[0080]Next, in the heating furnace 4 after applying the above-mentioned coating liquid to the surface, lengthening glass fiber like Example 1 using the same device (device shown in drawing 1) as Example 1, It was made to dry at 150 \*\*, it heat-treated at 600 \*\* for 1 hour after forming in the fiber surface the coating layer which consists of a glass bead and a titania precursor (inside of the atmosphere), and the projection layer with photocatalyst ability was formed.

[0081]Thus, in the obtained filter material, the average thickness of 70 micrometers and a projection layer of the path of glass fiber was 11 micrometers. When the fiber surface was investigated by microscope observation, it turned out that many projections are formed.

Although strongly wiped away by KIMUWAIPU which wetted this filter material in isopropanol, the projection did not exfoliate.

[0082]The solidified body of the above-mentioned coating liquid was produced, and the X diffraction was performed about what was heat-treated at 600 \*\* for 1 hour (inside of the atmosphere). The chart is shown in drawing 5. Thereby, it turned out that anatase type titania is crystallizing.

[0083]Example 4 (the 2nd method)

3.66 kg of isopropanol was mixed with 1.27 kg of tetraethyl orthosilicate, and the hydrochloric acid water 110g of 0.15-mol [l. ] concentration was added gradually, and was agitated for 1 hour. 4.04 kg of titanium tetraisopropoxide was added to this solution, and it agitated for further 1 hour, and 3.70 kg of ethyl acetoacetate was added and it agitated overnight. Subsequently, after adding gradually the hydrochloric acid water 731g of 0.15-mol [l. ] concentration, and a mixed solution with 15.00 kg of isopropanol to this solution and agitating them in it for 3 hours, 1.5 kg of glass beads with a mean particle diameter of 5 micrometers were made suspended, and 30 kg of coating liquid was prepared.

[0084]Next, the above-mentioned coating liquid was applied on the surface of glass fiber, and the projection layer which heat-treats and has photocatalyst ability after desiccation was made to form using the device shown in drawing 6, as shown below.

[0085]The glass fiber 3 which said drawing 1 for drawing 6 to make the projection layer which has photocatalyst ability on the surface of a fiber forming is a schematic diagram of a different device, and was sent out from the delivery roll 6, After passing through the inside of the coating liquid 8 accommodated with the coating cistern 7 and applying coating liquid to the surface, in the heating furnace 4, it was heat-treated at 600 \*\* after desiccation at 150 \*\* for 1 hour (inside of the atmosphere), and, subsequently was rolled round by the winding roll 5.

[0086]Thus, in the obtained filter material, the average thickness of 40 micrometers and a projection layer of the path of glass fiber was 6 micrometers. When the fiber surface was investigated by microscope observation, it turned out that many projections are formed. Although this filter material was strongly wiped away by KIMUWAIPU which carried out humidity by isopropanol, the projection did not exfoliate.

[0087]It turned out that the solidified body of the above-mentioned coating liquid is produced, and anatase type titania is crystallizing about what was heat-treated at 600 \*\* for 1 hour (inside of the atmosphere) when an X diffraction is performed.

[0088]Example 5 (the 2nd method)

1.5 kg of glass beads with a mean particle diameter of 30 micrometers were made suspended to 28.5 kg of titania thin film coating liquid (type whose anatase type titania deposits by the Nippon Soda Co., Ltd. make, trade name:NTi-500, and heat treatment), and 30 kg of coating liquid was prepared.

[0089]Next, after applying the above-mentioned coating liquid to the surface like Example 3, lengthening glass fiber, it heat-treated at 500 \*\* after desiccation at 150 \*\* for 1 hour (inside of the atmosphere), and the projection layer with photocatalyst ability was formed.

[0090]Thus, in the obtained filter material, the average thickness of 50 micrometers and a

projection layer of the path of glass fiber was 31 micrometers. When the fiber surface was investigated by microscope observation, it turned out that many projections are formed. Although strongly wiped away by KIMUWAIPU which wetted this filter material in isopropanol, the projection did not exfoliate.

[0091]It turned out that the solidified body of the above-mentioned coating liquid is produced, and anatase type titania is crystallizing about what was heat-treated in the atmosphere at 500 \*\* for 1 hour when an X diffraction is performed.

[0092]Example 6 (photocatalyst filter)

The photocatalyst filter shown in drawing 7 was produced using about 90,000 glass fiber in which the projection layer which was obtained in Example 3, and which has photocatalyst ability in the surface was provided, and the photocatalyst filter apparatus as shown in drawing 8 was constituted. Drawing 7 is a detail view of a photocatalyst filter, and it is the figure with which the elevation looked at (a) and (b) looked at a side view and (c) from the upper part. Drawing 8 is a mimetic diagram of a photocatalyst filter apparatus, and comprising the photocatalyst filter 11, the fan 12, and the ultraviolet ray source lamp 13 provided with the reflector 14 is shown.

[0093]This photocatalyst filter apparatus was put into the container with a content volume of 20 l., decomposition treatment of acetaldehyde (gas) was performed, and catalytic activity was evaluated. The ultraviolet ray intensity in the fiber end set initial concentration of  $1.0 \text{ mW/cm}^2$  and acetaldehyde gas to about 2000 ppm. A graph shows the relation between lapsed time and acetaldehyde concentration to drawing 9. In drawing 9, a black dot seal is a case where a photocatalyst filter apparatus is used, and a white round mark is a case where this device is not used.

[0094]This drawing 9 shows that the used photocatalyst filter has the outstanding photocatalyst effect.

[0095]

[Effect of the Invention]According to this invention, the filter material which can provide cheaply the highly efficient filter which has the photocatalyst ability used suitably for water treatment, an environmental clean-up, etc. can be manufactured efficiently.

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[Translation done.]

\* NOTICES \*

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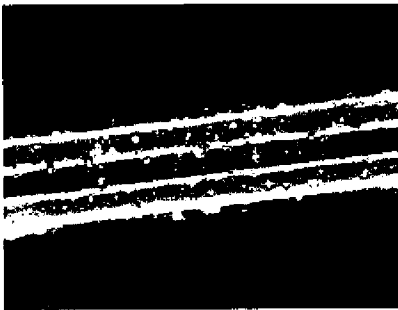
- 1.This document has been translated by computer. So the translation may not reflect the original precisely.
- 2.\*\*\*\* shows the word which can not be translated.
- 3.In the drawings, any words are not translated.

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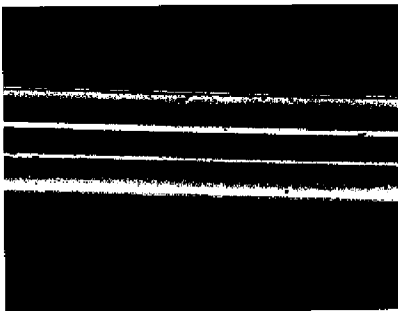
## DRAWINGS

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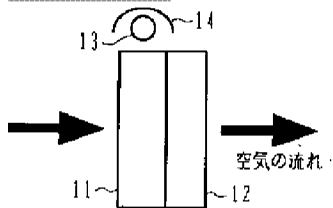
[Drawing 2]



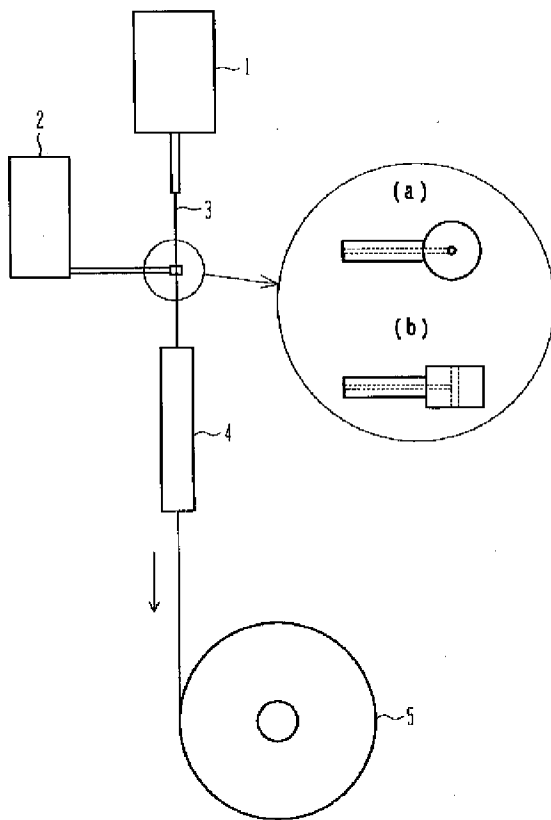
[Drawing 3]



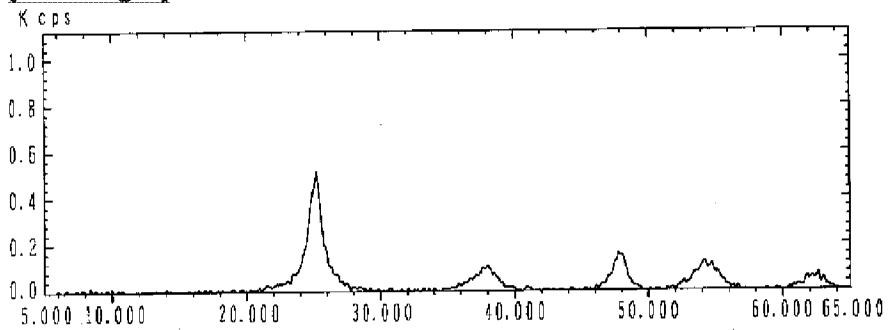
[Drawing 8]



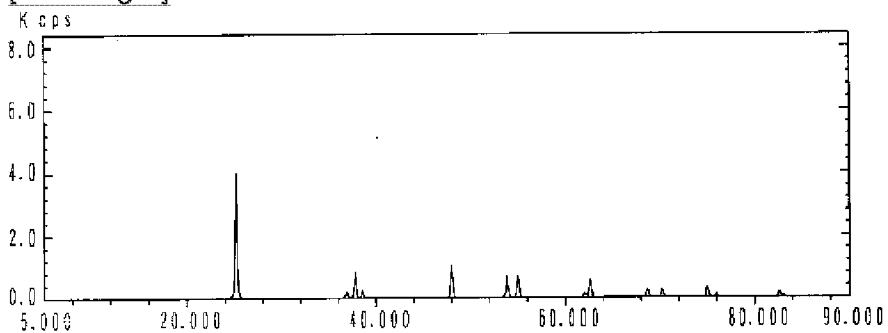
[Drawing 1]



[Drawing 4]

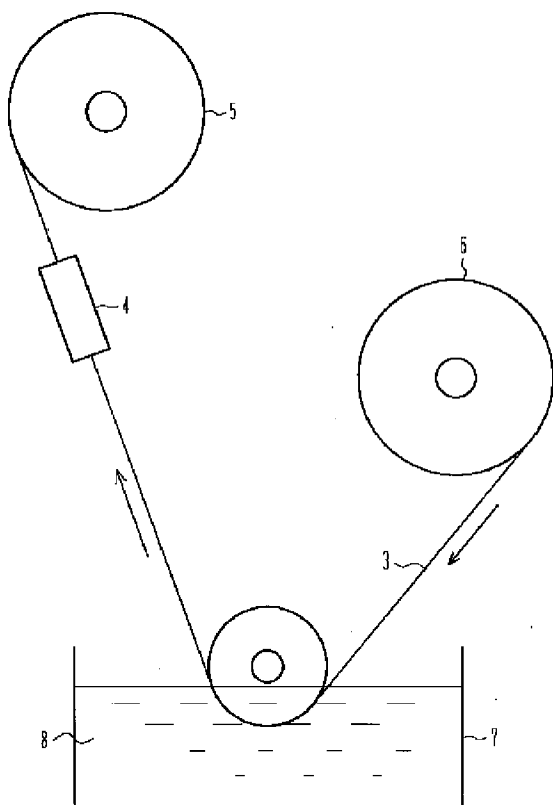


[Drawing 5]

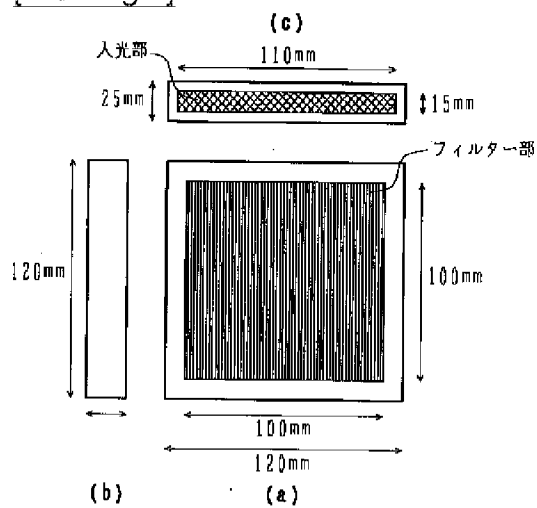


[Drawing 6]

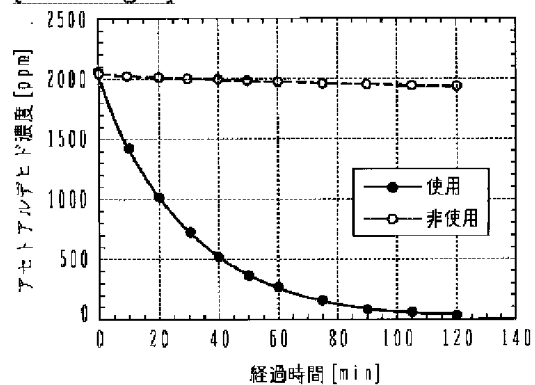




[Drawing 7]



[Drawing 9]



[Translation done.]